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**REPORT**

**MRL-R-778**

THE LOW TEMPERATURE ANNEALING OF 7.62 mm BRASS CARTRIDGE CASES:  
STRESS CORROSION SUSCEPTIBILITY

David S. Saunders

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REPORT

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ABSTRACT

> This report outlines an investigation into the effects of hardening during low temperature annealing on the stress corrosion susceptibility of brass cartridge cases. The low temperature annealing occurs in the lower walls during the mouth annealing process in the production of 7.62 mm cartridge cases.

In this work cartridge cases were taken from the production line prior to the mouth anneal and then annealed in an oil bath at 200°C or 250°C for a range of times. These cases were then exposed to an ammonia environment for 4 h. The tests showed that there was no evidence of enhanced stress corrosion susceptibility at higher wall hardness resulting from the artificial low temperature annealing. The degrees of cracking in the lower walls of the cartridge cases were found to decrease with increasing times of annealing with no evidence of cracking for times in excess of 6 h at 250°C. However, the trends in the results for the cases annealed at 200°C were less obvious. Reasons for this are put forward.

The hardening in the cartridge cases on low temperature annealing is explained in terms of modern theories of recovery in cold worked brasses.

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POSTAL ADDRESS: Chief Superintendent, Materials Research Laboratories  
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(Continued)

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## THE LOW TEMPERATURE ANNEALING OF 7.62 mm BRASS CARTRIDGE CASES:

### STRESS CORROSION SUSCEPTIBILITY

#### 1. INTRODUCTION

It has been reported [1] that in the manufacture of the 7.62 mm cartridge case the process of mouth annealing produces an increase in the hardness of the lower wall near the base region. A drawing of the 7.62 mm cartridge case is shown in Figure 1. The lower wall receives only a low temperature anneal which results from a thermal gradient from the mouth of the case to the base that is established during the mouth annealing heat treatment. During this production process a temperature range of 550°C at the mouth to 200°C at the base is expected.

The question was raised as to whether this increase in hardness (if real) was accompanied by an increased susceptibility to stress corrosion beyond the sensitivity of the mercurous nitrate test [2]. For example, brasses containing much lower residual stresses than those necessary to produce cracking in a mercurous nitrate solution may fail in an ammoniacal atmosphere [3].

This report describes work carried out to investigate the hardening phenomenon in cold-worked cartridge brass on low temperature annealing, and to assess any consequent changes in susceptibility to stress corrosion cracking in 7.62 mm cartridge cases.

#### 2. EXPERIMENTAL

The hardening phenomenon was examined in both cartridge cases and cold rolled strip. Tests for stress corrosion susceptibility of the cartridge cases were carried out using an ammonia environment which other workers have shown to be more sensitive than the mercurous nitrate test [3]. The severity of cracking would be expected to be related to the hardness and/or the level of residual stress in the lower walls of the cartridge cases. In order to produce a range of hardness states in the walls of the cartridge cases artificial low temperature annealing heat treatments were used.

## 2.1 Details of Test Specimens

- (i) strips reduced 86, 60 and 50 per cent in thickness by cold rolling, i.e. 86, 60 and 50% CR.
- (ii) cartridge cases which had been taken from the production line immediately prior to the mouth anneal. These cases are referred to as the cold worked (CW) cases throughout this report.
- (iii) cartridge cases which had been mouth annealed.

The composition specification of the material [4], is given in Table 1.

T A B L E 1

COMPOSITION OF BRASS FOR 7.62 mm CARTRIDGE CASES\*  
(wt %)

	Cu	Mo	Fe	Bi	Ni	Sn	As	Sb	Al	Other Elements (each)	Zn
Max	72.0	0.02	0.05	0.004	0.1	0.03	0.01	0.005	0.004	0.005	rem.
Min	69.0										

\* Taken from Ref. 4

## 2.2 Heat Treatments

The heat treatments used in the experimental work are summarised in Table II.

T A B L E II

HEAT TREATMENT OF TEST SPECIMENS

Material	Designation used in the text	Heat Treatment	Comments
cold rolled strips (86, 60 and 50% CR)	CRA	low temp. anneals at 200°C and 250°C for a range of times	to give a range of hardness states
cold worked cartridge cases	CW and CWA (after annealing at 200°C and 250°C)	" "	to give a range of hardness states in the walls of the cases
mouth annealed cartridge cases	MA	no heat treatment; as received from the production line	for control experiments

The low temperature annealing heat treatments were carried out at 200°C and 250°C. The procedure was based on the earlier experiments reported by Mackintosh [5], Appendix I, which showed greatest hardening at 250°C.

The cold rolled strips and the cold worked cartridge cases were annealed by complete immersion in an oil bath controlled to  $\pm 1^\circ\text{C}$  with heat treatment times varying from 5 min to 18 h at each heat treatment temperature. Following the low temperature anneals, the hardness of each test piece was measured using a Frank hardness testing machine with a 2.5 kg load. Hardness measurements were taken at points 5 mm from the mouth (mouth region) and 3 mm from the base (lower wall region). The results were then plotted as aging curves.

### 2.3 Stress Corrosion Tests on Cartridge Cases

#### (1) Mercurous Nitrate Test

Three cold worked cases (CW) and three mouth annealed cases (MA) were subjected to the mercurous nitrate test set out in Australian Army Specification 293 [2]. These tests were used as a standard and the sensitivity of the ammonia environment test was compared against this standard.



### *(ii) Ammonia Environment Test*

An ammonia environment test was based on the experimental procedure reported by Jamieson and Rosenthal [6]. The experimental system used in the present work consisted of a large glass desiccator vented to the atmosphere through a capillary tube; in the lower reservoir of the desiccator was placed 1.2 L of ammonia solution (S.G. 0.941). The low temperature annealed cases (CWA), the cold worked cases (CW), and the mouth annealed cases (MA) were placed on a porcelain plate above this ammonia solution. The system was kept at about 20°C. After the cases had been exposed to the ammonia environment for a period of 4 h, they were removed, pickled in a 40% HNO<sub>3</sub> solution for 15 s, washed in alcohol and then the severity of cracking assessed.

### *2.4 Metallographic Examination*

Cartridge cases which had been exposed in the ammonia environment were examined metallographically to assess the extent of cracking through the walls. Sections were also taken through rolled sheets and other low temperature annealed cases to observe any microstructural changes arising from the heat treatment of the cold worked material.

## 3. RESULTS

### *3.1 Hardness Changes on Annealing*

The hardness data are plotted as aging curves in Figures 2(a,b) and 3(a,b). The hardness increase on low temperature annealing is quite small, but readily measurable and reproducible.

The results in Figure 2(a,b) were obtained from cold rolled strip (CRA). These data are not truly representative of the hardness increases in cartridge cases arising from the mouth annealing process because the % cold work in the lower walls of a cartridge case is lower than that in the rolled strips used in this work. Thus the hardness increases observed in Figure 2(a,b) possibly represent the extreme condition of hardening on low temperature annealing.

The results for the low temperature annealing of the cold worked cartridge cases (CWA) are plotted in Figure 3(a,b) together with the average lower wall hardness attained as a result of the mouth annealing process. This average hardness level is located at the 2 min mark for convenience. In fact, the case is in the induction furnace for mouth annealing for only about 15 s, but the point on the figure arbitrarily allows for the slower cooling of the thick-walled base region.

### *3.2 Stress Corrosion Tests on Cartridge Cases*

#### *(i) Mercurous Nitrate Test*

The testing of stress corrosion susceptibility of the mouth annealed cartridge cases (MA) and the cold worked cartridge cases (CW) using the mercurous nitrate test was inconclusive, as expected from the report of

earlier work [1]. None of the three mouth annealed cases exhibited cracking and only one of the three cold worked cases showed a crack in the lower wall region. This result was inconclusive because no clear distinction between the mouth annealed and cold worked cases and their respective sensitivities to stress corrosion cracking could be made.

### (ii) Ammonia Environment Test

The testing of stress corrosion susceptibility using the ammonia environment showed that both the cold worked (CW) and mouth annealed (MA) cartridge cases crack in the lower wall regions, although cold worked cases generally showed more severe cracking Figure 4(a,b).

Those cold worked cases which were also given low temperature anneals at 250°C (CWA) showed decreasing severity of cracking (see next paragraph) with increasing time at the annealing temperature. The cold worked cartridge cases which had been heat treated for 6 h and 18 h at 250°C exhibited no cracking.

The trends in the results from those cold worked cases which were given low temperature anneals at 200°C (CWA) were less obvious. Cracking was observed in the lower walls of the cases, with little discernible further decrease in its severity after the initial decrease following the 10 min low temperature anneal. The cold worked case annealed for 13 h seemed to have cracking which was more severe than those cases given shorter low temperature anneals, and this cracking was non-uniformly distributed around the lower wall. The result for this case was considered to be anomalous.

The severity of cracking was a qualitative assessment and relied on observations using a binocular microscope. For example Figure 4(c) shows the severity of cracking for a range of low temperature annealing times at 250°C. This illustrates, however, that it was possible to rank decreasing severity of cracking, and that this generally corresponded to increasing annealing times.

The stress corrosion cracks caused by the ammonia environment were generally completely through the walls of the cartridge cases and Figure 5 illustrates that they were largely intergranular and often branched. Some general surface attack by the ammonia environment was also observed.

### 3.3 Microstructural Changes on Annealing

The microstructural changes on annealing at low temperatures proved difficult to study by optical microscopy. However, partial recrystallisation occurred near the mouths of the cold worked cases (see Figure 6) and with the 86% cold rolled strip, on annealing at 250°C for times in excess of 6 h, (specimens CWA and CRA).

No recrystallisation was observed in the lower walls of the mouth annealed cases and those cold worked cases subjected to low temperature anneals.

No evidence of recrystallisation was observed in the mouth regions of those cold worked cartridge cases annealed at 200°C for times up to 18 h.

#### 4. DISCUSSION

##### *4.1 Hardness Changes on Annealing*

The hardness increase in cold rolled brass on low temperature annealing is widely reported [1,7,8], but the effect has not been satisfactorily explained. Harrington and Jester [8] suggested that the effect may be the result of the precipitation of  $\beta$  within  $\alpha$  due to the high residual stresses. Reviewers of their work [9] considered this to be unlikely. The work of Clarebrough et al. [10,11] and of Sato [12] investigated the release of stored energy from deformed Cu/Zn alloys. This work has been reviewed by Bever et al. [13]. Clarebrough et al. [10] reported a slight increase in hardness in 69 Cu/31 Zn alloy deformed at room temperature and anisothermally annealed. A hardening peak was observed at approximately 220°C. The release of stored energy from deformed brass appeared as three distinct stages; the hardening occurring during the second stage. The peak in rate of energy release and increase in hardness during this stage was considered to be a result of the return of short range order destroyed during cold work. Clarebrough et al. [11] considered that these changes in properties during annealing were complex and that some dislocation rearrangement must also take place. Recent work reported by Huber and Hatherly [14] suggested, however, the growth of "recovery twins" could account for the release of stored energy from 200-250°C reported earlier [10]. Their work on 90% cold rolled 70 Cu/30 Zn alloy showed that "recovery twins" formed at temperatures as low as 200°C and hence could largely contribute to the peak in stored energy release observed by Clarebrough et al. Hardening associated with such recovery mechanisms may be due to the large number of fine twin boundaries in the recovering microstructure [15].

The hardness increases observed in the present work were small for all cold worked cartridge cases subjected to short low temperature annealing heat treatments (CWA). The hardening in the lower walls of the cartridge cases was particularly small, as illustrated in Figure 3(a,b). This was the result of the relatively low levels of cold work in these regions of the cartridge cases as against the mouth regions where the hardness increases on low temperature annealing were more pronounced.

The final, significant decrease in hardness after 250°C low temperature annealing of heavily cold rolled brass (CRA) may largely be attributed to recrystallisation Figure 2(b). On the other hand, the hardness changes observed in these cold rolled specimens annealed at 200°C (CRA) suggest that no recrystallisation, detectable by optical microscopy, has occurred over the times used in these experiments, Figure 2(a). This heat treatment may be on the lower limit of that for the recovery process to become operative and hence may largely account for the inconclusive results obtained in the stress corrosion tests of cartridge cases which were annealed at 200°C.

The observations of the hardness changes on low temperature annealing in the present work and that reported by Mackintosh [5] were consistent with those reviewed by Bever et al. [13].

#### 4.2 Stress Corrosion Tests on Cartridge Cases

The present work has shown that there does not appear to be an increased susceptibility to stress corrosion cracking through the peak in hardness as a result of the low temperature annealing of cold worked cartridge cases (CWA). Indeed, despite measurable increases in hardness in the lower walls of both the mouth annealed cases (MA) and the low temperature annealed cold worked cases (CWA), cracking was less severe than that of cases in the cold worked (CW) condition. Such observations are made on test pieces which have been subjected to the most sensitive test presently available and those regions where cracking normally occurs have only very small changes in hardness as a result of low temperature annealing heat treatments.

The experimental evidence suggests that the decrease in susceptibility to stress corrosion cracking in the 7.62 mm cartridge cases with increase in time of low temperature annealing is associated with a decrease in residual stress levels, possibly as a result of the rearrangement and loss of dislocations during the recovery process. This is particularly significant for brass containing a high level of cold work where recrystallisation takes place on extended times of low temperature annealing, see 4.3.

Attempts to measure residual stresses in the cartridge cases by X-ray techniques during this work were unsuccessful. There may be a number of reasons for this. For example, Witt et al. [16] suggest that residual stresses across individual grains are not uniformly distributed. However, it is more likely that the texture developed during the cold drawing processes tends to modulate the X-ray data.

#### 4.3 Microstructural Changes on Annealing

The optical microscopy of the mouth regions of the cold worked cartridge cases and sheet specimens annealed at 250°C for long times showed evidence of partial recrystallisation, and this is believed to account for the rapid decrease in hardness after 3 h, (Figure 3(b)). This result is consistent with the work of Clarebrough et al. [10].

#### 4.4 A Comment on the Ammonia Environment Stress Corrosion Test

It has been shown that, to obtain reproducibility with the ammonia environment stress corrosion test, it is necessary to control the initial concentration of the ammonia solution, the volume of the solution relative to the container, and the temperature of the system [6]. Thus, for constant exposure times, it would be expected that the vented chamber test used in this work would give reproducible results despite the fact that the system does not control the partial pressure of each gas independently.

It has been reported that a minimum threshold stress is necessary to induce cracking in a mercurous nitrate solution [17,18]; however, it is known that the ammonia environment test is more sensitive [3]. Despite this obvious advantage no ammonia environment test is accepted as a standard test for stress corrosion susceptibility of brass. This may be due to the fact that extensive world-wide experience has shown that if no cracking occurs in the mercurous nitrate solution then no cracking occurs in service. Furthermore, the mercurous nitrate test is simple to use and hence accepted as a factory test.

## 5. CONCLUSIONS

The ammonia environment test is a very sensitive test for determining the susceptibility of brass to stress corrosion cracking, yet it did not appear to indicate an increase in severity of cracking through the hardening peak in cold worked cartridge cases which had been annealed at low temperatures. Thus it appears that, within the limitations of the evaluation techniques used in this work, there was no measurable increase in sensitivity to stress corrosion cracking as a result of the mouth annealing heat treatment used in the production of 7.62 mm cartridge cases.

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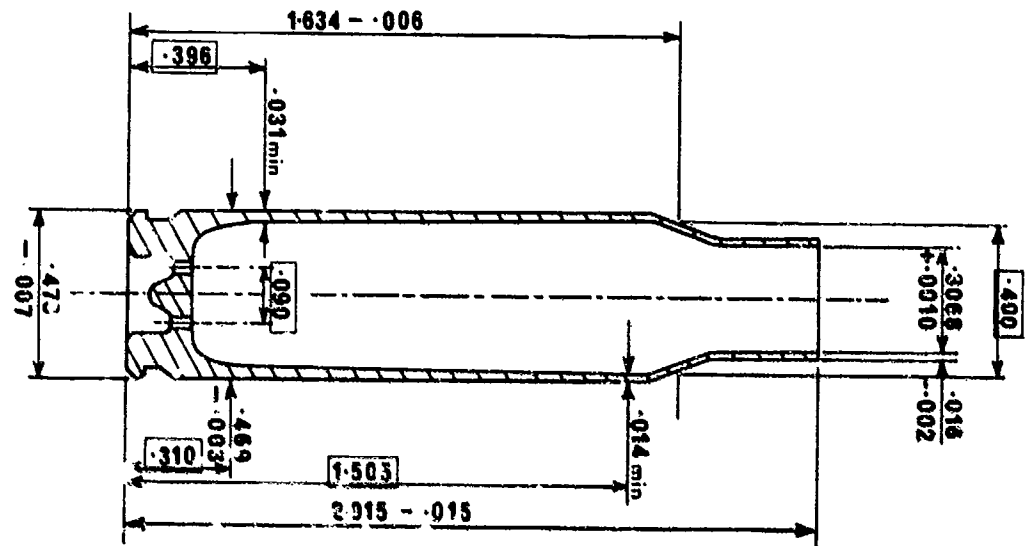
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APPENDIX I

EFFECT OF LOW TEMPERATURE ANNEALING ON THE MECHANICAL

PROPERTIES OF COLD WORKED 70/30 BRASS, REF. [5]

Property	Condition	Per cent Reduction by Rolling					
		25		60		80	
			% Change		% Change		% Change
Hardness HV 10	as received	142		186		204	
	250°C for 1 h	145	+ 2	196	+ 5	219	+ 7
	255°C " "	146	+ 3	193	+ 4	216	+ 6
	260°C " "	145	+ 2	194	+ 4	215	+ 5
0.2% Proof Stress MPa	as received	391.6		560.8		626.9	
	250°C for 1 h	386.5	- 1	578.0	+ 3	667.0	+ 6
	255°C " "	381.9	- 2	571.8	+ 2	623.5	- 1
	260°C " "	389.1	- 1	572.6	+ 2	628.4	0
UTS MPa	as received	431.0		603.9		698.4	
	250°C for 1 h	441.9	+ 3	631.7	+ 4	725.6	+ 4
	255°C " "	438.8	+ 2	625.4	+ 3	698.9	0
	260°C " "	438.9	+ 2	614.9	+ 1	692.6	- 1
% El. on 50 mm	as received	20		4.2		3.5	
	250°C for 1 h	22.5	+ 12.5	2.2	- 48	2.5	- 22
	255°C " "	24.5	+ 22.5	3.7	- 12	3	- 14
	260°C " "	23.0	+ 15	4	- 5	3.2	- 9

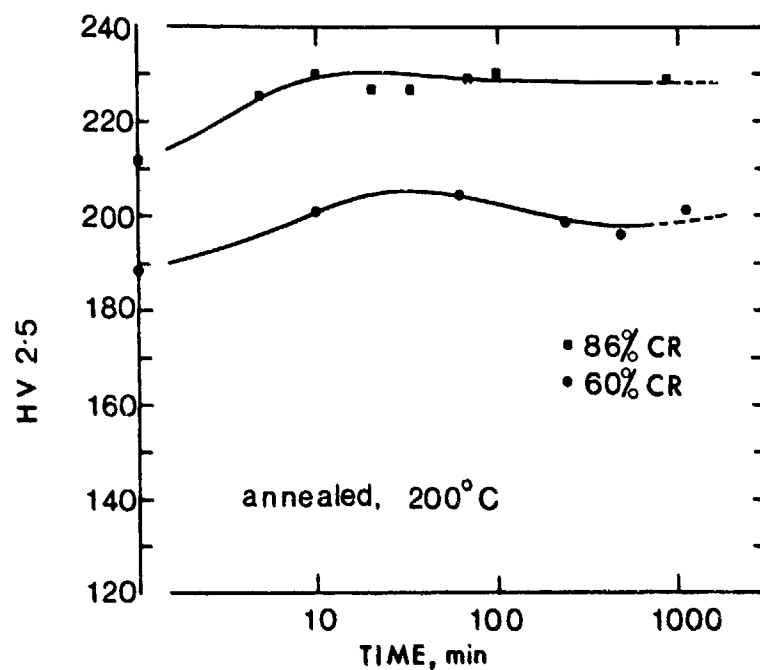


dimensions in inches

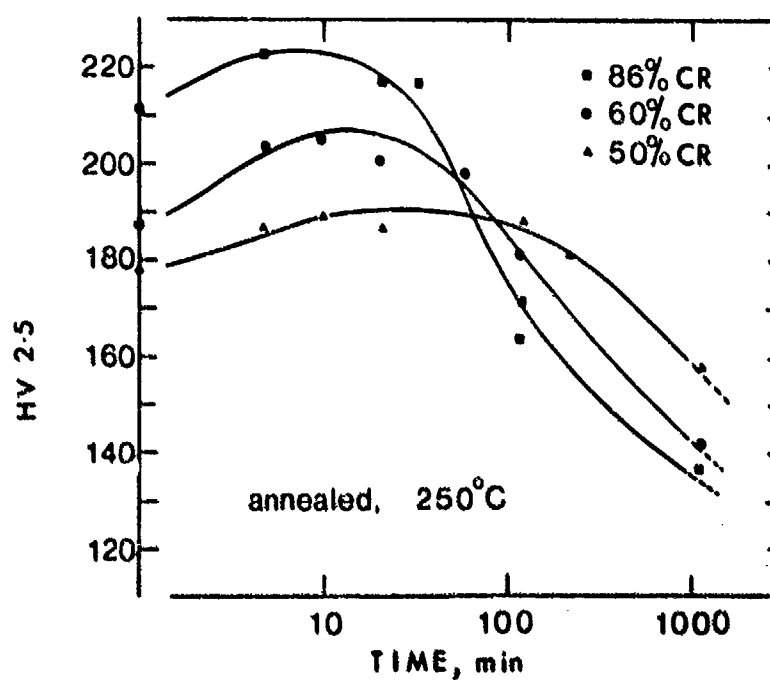
brass, to Army (Aus) 218

FIG. 1 - The 7.62 mm Cartridge Case, as finished.



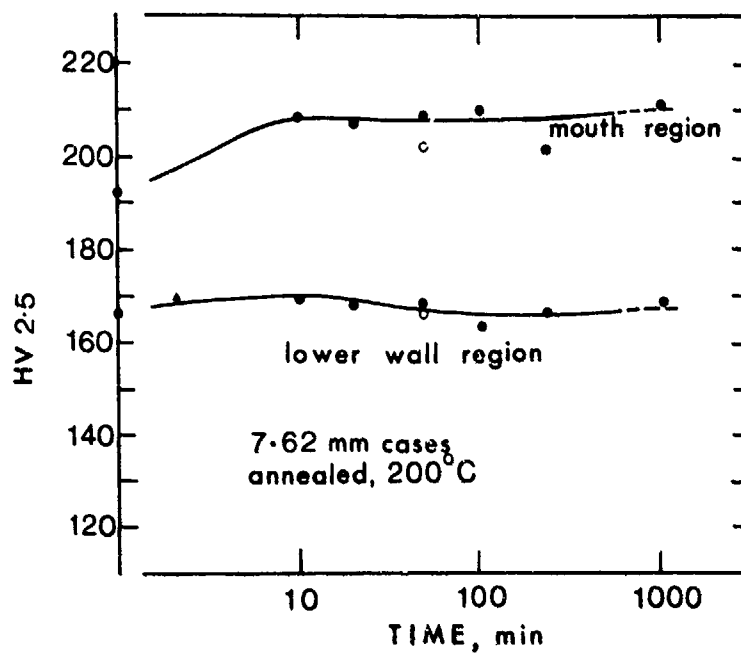


(a)

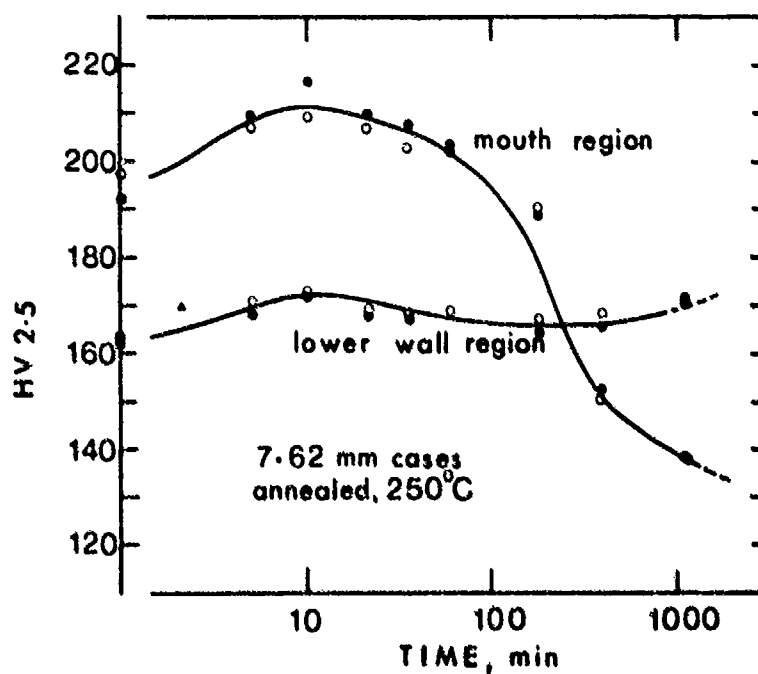


(b)

FIG. 2 - Graph of hardness (HV2.5) plotted against time of low temperature annealing for cold rolled strips, (a) 200°C anneal; (b) 250°C anneal.



(a)



(b)

FIG. 3 - Graph of hardness (HV2.5) plotted against time of low temperature annealing for cartridge cases initially in the cold worked condition.  
 (a) 200°C anneal; (b) 250° anneal.  
 Hardness measurements taken at points 5 mm from the mouth (mouth region) and 3 mm from the base (lower wall region). The symbol  $\Delta$  represents the average hardness of the lower wall region after mouth annealing, arbitrarily placed on the figure at 2 min.



(a) cold worked (CW)



(b) mouth annealed (MA)



5 min



3 h



6 h

(c) low temperature annealed at 250°C (CWA)

FIG. 4 - Stress corrosion cracking in the lower walls of the cartridge cases induced by exposure to an ammonia environment for 4 h.



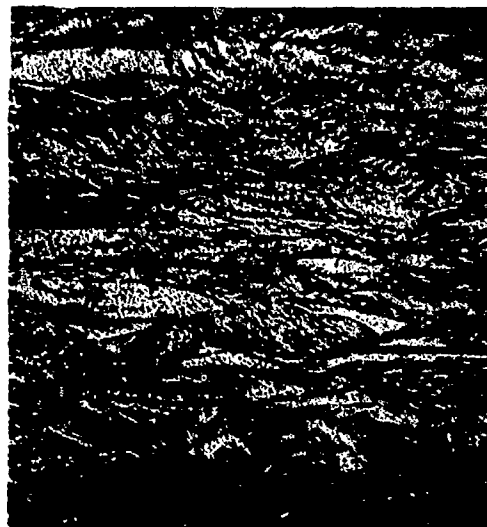
(a) cold worked (CW) 200X



Outer wall of the  
cartridge case

(b) mouth annealed (MA) 100X

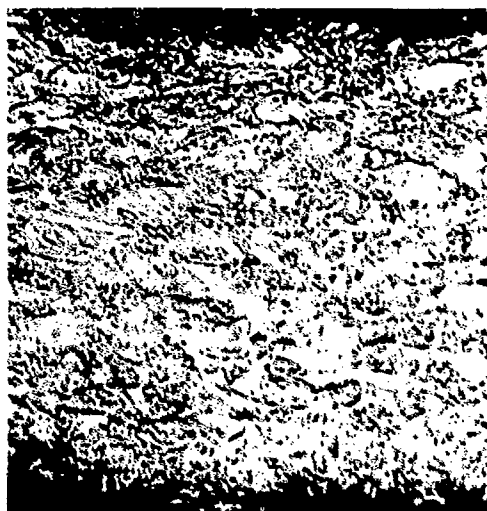
FIG. 5 - Stress corrosion cracks in the lower walls of 7.62 mm cartridge cases induced by exposure to an ammonia environment for 4 h. Cracking in both cases is largely intergranular.



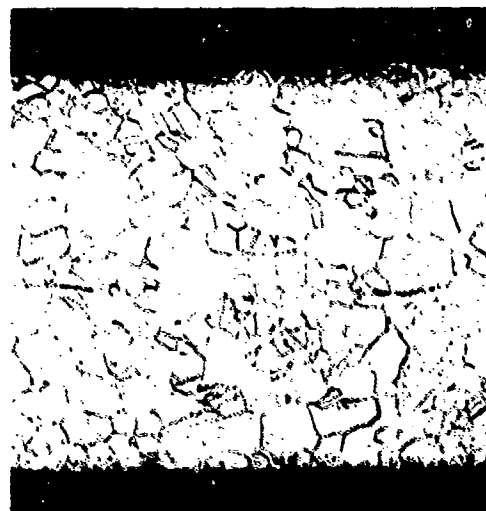
(a) cold worked (CW)



(b) cold worked and low  
temperature annealed  
for 18 h at 200°C (CWA)



(c) cold worked and low  
temperature annealed  
for 18 h at 250°C (CWA)



(d) mouth annealed (MA)

FIG. 6 - Microstructures of the mouth regions of 7.62 mm cartridge cases.  
x 250

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The Director, Defence Scientific Information and Documentation  
Centre, Delhi, India  
Colonel B.C. Joshi, Military, Naval and Air Adviser, High  
Commission of India, Red Hill, A.C.T.  
Director, Defence Research Centre, Kuala Lumpur, Malaysia  
Exchange Section, British Library, Lending Division, Yorkshire,  
England  
Periodicals Recording Section, Science Reference Library, British  
Library, Holborn Branch, London, England  
Library, Chemical Abstracts Service, Columbus, Ohio, U.S.A.  
INSPEC: Acquisition Section, Institution of Electrical Engineers,  
Hitchin, Herts, England  
Overseas Reports Section, Defence Research Information Centre,  
Orpington, Kent, England  
Science Information Division, Department of Scientific and  
Industrial Research, Wellington, New Zealand.